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Analysis of Volatile Compounds from Supercritical Extracted Soybeans by Headspace Gas Chromatography and Thermal Desorption of a Polymer Adsorbent

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Abstract: Soybean flakes were extracted with supercritical carbon dioxide to produce a solvent-free, good-quality soybean oil. Volatile compounds from the supercritical fluid extracted (SFE) oil and from a hexane-extracted crude soybean oil were analyzed by dynamic headspace gas chromatographic methods to determine qualitative differences between the extraction methods. The major difference in the volatile profiles was the higher concentration of hexane in the solvent-extracted oil. During the SFE process, volatile compounds were trapped on a porous polymer trap attached at the exhaust port of the SFE apparatus. The volatile profile obtained from the sorbent trap was found to be similar to the headspace profile from the SFE/soybean oil removed during the same extraction. In addition, crude soybean oil was heated in a stirred reactor and the volatiles, which were stripped by supercritical carbon dioxide in an attempt to improve oil properties, were collected on sorbent traps and analyzed by the above method for comparison. The described methodology permits the characterization of volatiles and semivolatiles in SFE soybean oil and can be used to monitor the extraction and quality of the resultant oil.

Key words: hexane-extracted, supercritical extracted, GC/MS, soybean oil, Tenax.

INTRODUCTION

Soybeans extracted with supercritidal carbon dioxide ($SC\text{-}CO_2$) yield an oil which is comparable to hexane-extracted degummed oil devoid of any solvent residue (Stahl *et al* 1980; Friedrich *et al* 1982; List and Friedrich 1985; List and King 1989). In addition, the supercritical fluid extraction (SFE) process has been shown to yield both an oil and meal that are improved in color and equivalent in flavor to those obtained from conventional extraction and refining processes (Friedrich and List 1982; Friedrich and Pryde 1984; Christianson *et al* 1984; List *et al* 1984; Eldridge *et al* 1986). Smelling of the decompressed CO_2 stream as it exits the extractor suggests the presence of many odoriferous components which are being removed during the extraction process.

Volatile components from a variety of samples, such as milk products, vegetable oils and floral specimens, have been trapped on porous polymer adsorbents to collect and concentrate the volatile analytes (Mills 1986; Selke and Frankel 1987; Raghavan *et al* 1989). In addition, on-line collection of pesticides onto a sorbent trap has been accomplished using SFE (Patt *et al* 1992).

The aim of this work was to use SFE to analyze lipid oxidation volatiles, to compare the volatiles in SFE-derived and hexane-extracted soybean oils and to examine a new experimental thermal processing procedure.

EXPERIMENTAL

Soybeans were cracked, dehulled and flaked; 60 g of the flakes were extracted with $SC\text{-}CO_2$ using a laboratory-built extractor (King *et al* 1989). The apparatus was

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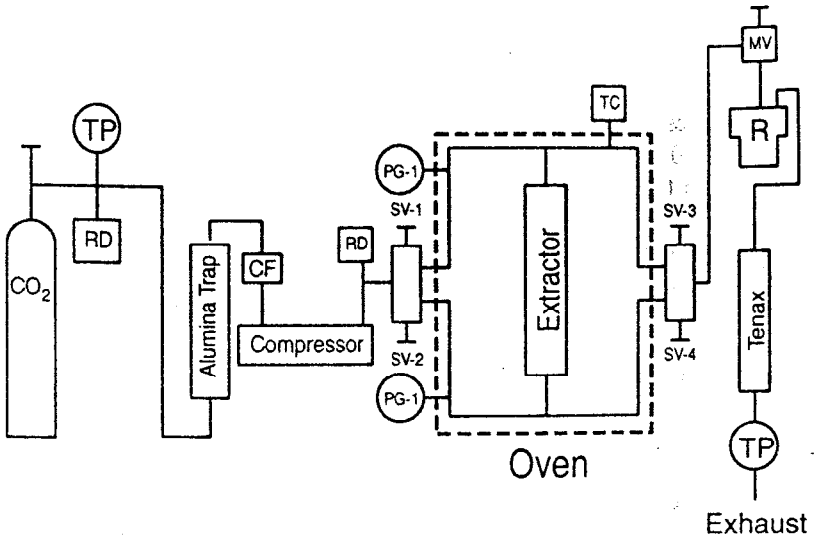
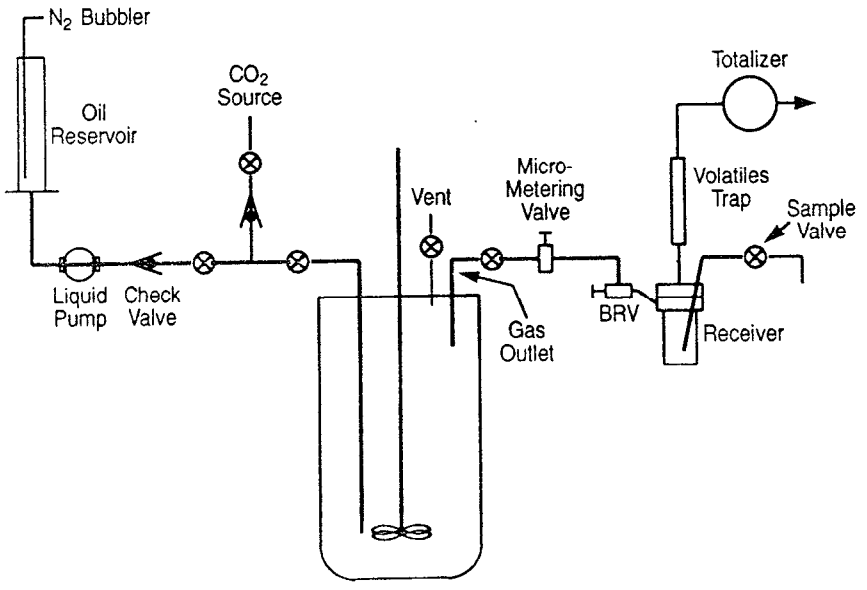


Fig 1. Diagram of SFE apparatus with Tenax trap at the exit port of the receiver. TP, total pressure gage; RD, rupture disk; CF, filter; PG, pressure gage; SV, shut-off valves; TC, thermocouple; MV, micro-metering valve; R, receiver; Tenax, trap filled with Tenax sorbent; TP, gas totalizer.

modified by the addition of a trap (18 cm × 6 mm id) containing 0·8 g Tenax TA (500–840 μm) sorbent (Alltech Associates, Inc, Deerfield, IL, USA) placed at the exit port of the collection vessel to collect the volatiles generated during the extraction (Fig 1).

The Tenax was conditioned before use to remove any interfering absorbed contaminants. The traps were heated for 30 min at 200°C with a helium flow of 50 ml min^{-1}. The traps were then placed inside a supercritical extraction apparatus (King *et al* 1989) for 30 min and subjected to extraction at 383 kPa and 50°C. After these conditioning processes, the traps were placed into tubes and tightly sealed to prevent any impurities from adsorbing onto the Tenax.

During the extraction of the oil, the CO_2 was decompressed to atmospheric pressure at the exit port of the collection vessel, where the flow rate of the CO_2 was monitored at 3 liters min^{-1} by a gas totalizer. A Tenax trap at ambient temperature was in position at the start of the extraction process and changed after passage of 20–30 liters of CO_2 through the trap. A larger tube filled with 6 g of Tenax was attached in series with the first trap to determine if there was breakthrough of the more volatile compounds during the sampling pro-



cedure. Samples of oil were removed from the collection vessel as the traps were changed throughout the entire extraction period. Extraction conditions were varied to examine the effect on the volatiles generated. The first extractions were conducted at 50°C and 383 kPa the second at 80°C and 479 kPa, and the third at 50°C and 96 kPa.

Crude soybean oil was also treated with SC-CO_2 in a high-pressure stirred autoclave as shown in Fig 2. Soybean oil (150 ml) was added to the vessel which was then filled with CO_2. The vessel was heated to 240°C and the pressure maintained at 96 kPa with the aid of a micro-metering valve. Soybean oil was metered through the system into the receiver vessel. Volatiles were collected on a Tenax trap located between the receiver and the gas totalizer. Oil samples were taken each time the Tenax traps were changed.

Volatile analysis of the SFE soybean oil and hexane-extracted soybean oil was accomplished by dynamic headspace sampling using a TekMar 4000 Headspace Concentrator (Cincinnati, OH, USA) in tandem with a Perkin Elmer Sigma 3B GC (Norwalk, CT, USA) and a Finnigan Mass Spectrometer, Model No. OWA 1050 (San Jose, CA, USA) (Selke and Frankel 1987). Helium was purged over the top of the oil at 100°C for 5 min as the volatile compounds were collected on Tenax; the volatiles were thermally desorbed at 150°C for 5 min onto a gas chromatograph (GC) capillary column (DB-1701, 30 m × 0·32 mm id; J & W Scientific, Folsom, CA, GC). The GC oven was held at −50°C during a 1 min splitless injection, then programmed to 250°C at 5°C min^{-1}. The injector temperature was 150°C. After 1 min, the eluent was split with a ratio of 50:1; the helium velocity through the column measured 26 cm s^{-1} at 100°C. The volatile compounds were measured using mass spectrometry in the electon impact (EI) mode; the ionization voltage was 70 eV over the mass range of 20–450 amu.

Analysis of the volatiles collected on Tenax traps at the exit of the extractor was accomplished by modifying the Tekmar Headspace Concentrator to thermally desorb the volatiles from the Tenax at 100°C during a 1 min splitless injection onto the GC capillary column. Identification of the compounds was made with the aid of Wiley/NBS mass spectrometer (MS) data system library which includes spectra of 131 000 compounds, and by using standard compounds.

TABLE 1
Headspace volatiles[a] from hexane-extracted and SFE soybean oils

Volatile compound	*Hexane extracted*	*SFE at 80°C and 479 kPa*
Ethanal	68·29	23·72
Pentane	190·01	66·43
Propanal	87·88	122·48
Ethanol	15·65	0·74
2-Methylpentane		93·69
Hexane	9653·93	19·26
Methyl cyclopentane	9·02	
Butanal	1·87	0·68
1-Propanol	0·10	0·33
2-Butanone	7·36	61·54
Heptane	145·50	16·28
2-Ethylfuran	16·88	37·73
2-Butenal	61·69	18·20
Pentanal	213·95	290·40
A butenol	0·09	0·03
1-Penten-3-one	0·33	0·01
2-Methylbutanal	19·07	27·75
Octane	15·51	2·35
2-Pentenal	88·16	47·80
1-Pentanol	100·76	188·86
Hexanal	2031·73	2982·03
A butendiol	0·07	0·08
1,3-Nonadiene	0·07	0·02
3-Pentenol	0·23	0·42
2-Hexenal	15·28	93·96
1-Hexanol	65·84	30·78
Heptanal	31·42	70·58
2-Pentylfuran	6·53	32·07
2-Heptenal	233·06	215·72
1-Octen-3-ol	3·32	37·26
Octanal	1·72	8·21
2t,4c-Heptadienal	2·35	32·26
2t,4t-Heptadienal	1·72	82·05
Hexanoic acid	5·34	103·59
2-Octenal	1·25	2·10
Nonanal	3·71	3·90
2-Nonenal	0·17	0·05
2t,4c-Decadienal	1·02	0·72
2t,4t-Decadienal	5·98	2·47
Other	2·55	0·46
Total	13 203·10	4532·32

[a] Values for volatiles are peak area × 10^3 and the average of two samples from different extractions.

RESULTS AND DISCUSSION

Comparisons were made between the volatile profiles from SFE-extracted soybean oil and hexane-extracted crude soybean oil. Identification of the compounds by GC–MS in Table 1 demonstrated that both types of extraction yielded typical volatile compounds in soybean oil (Frankel 1985). Identified components included C_2 to C_9 saturated aldehydes and the mono-unsaturated aldehydes, 2,4-heptadienal and 2,4-decadienal isomers. Also present were C_5 to C_8 saturated and unsaturated hydrocarbons, ethylfuran and pentylfuran, fatty acids and traces quantities of some ketones and alcohols. Many of the individual volatile components in

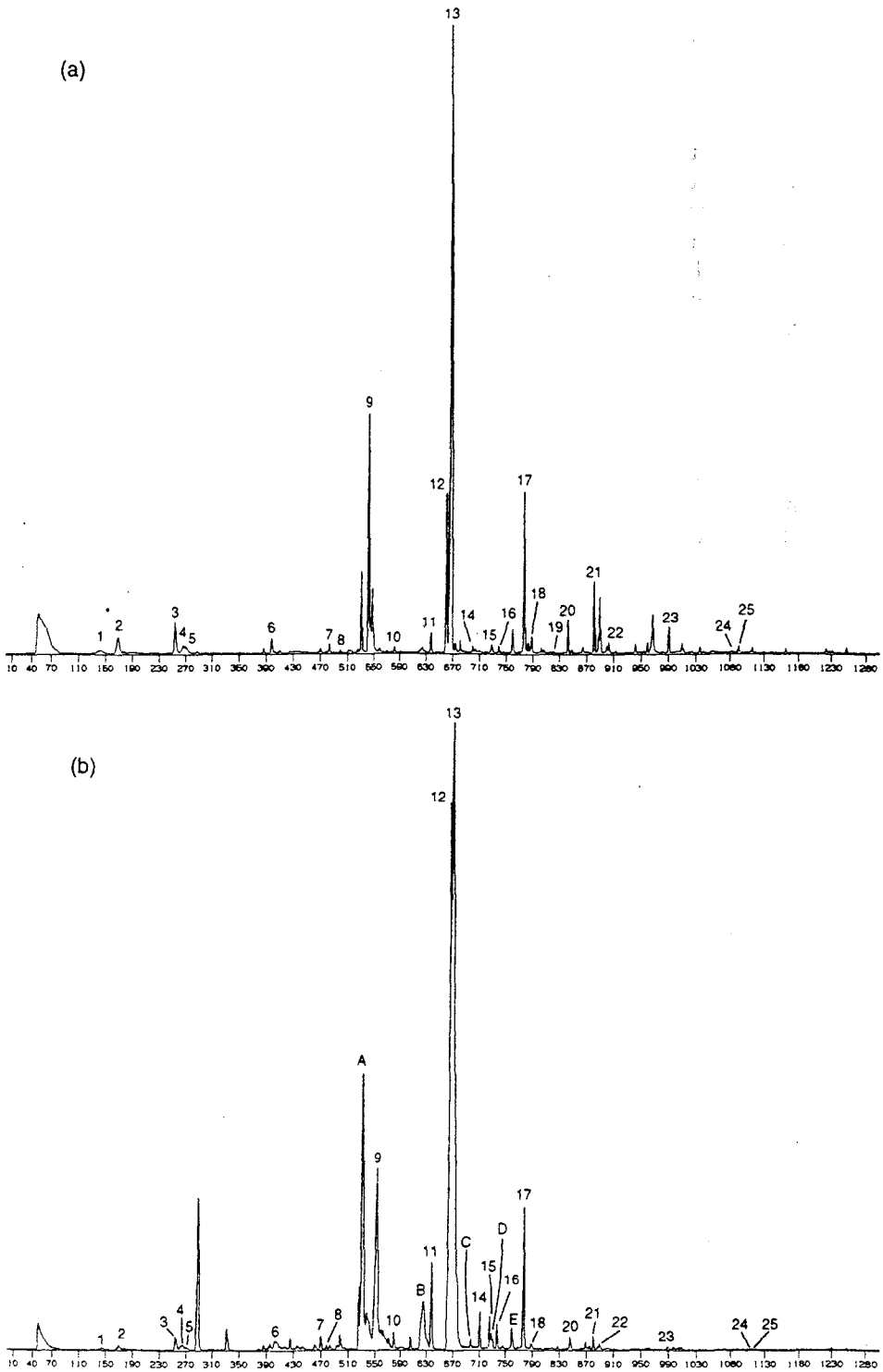


Fig 3. Total ion chromatogram of SFE headspace volatiles from (a) soybean oil (80°C and 479 kPa); and (b) Tenax trap volatiles. Peak numbers correspond to the compounds listed in Table 2. Identification of lettered peaks include A, acetic acid; B, 1,2-propandiol; C, pentanone; D, butyrolactone; E, ethyl benzene.

the SFE soybean oil at 80°C and 479 kPa were similar to the hexane-extracted oil as shown in Table 1. The solvent-extracted soybean oil had larger concentrations of hexane, methyl pentanes, and methyl cyclopentane; these compounds had smaller peak areas or were not present in the SFE oils. Apparently, the extraction solvent was not completely removed during the long evaporation step and was evident in the volatile analysis as a major component. The largest volatile peak in the SFE oil was hexanal which is an oxidation

product of linoleate (Selke *et al* 1980; Snyder and Mounts 1990). The peak areas of hexanal were comparable in both oils. Pentane, propanal, pentanal and 2-heptenal are also major components in both extractions. Ethanal, heptane and pentenal are at larger concentrations in the hexane-extracted oil, while propanal, 2-butanone, 2-hexenal and heptanal are larger in the soybean oil extracted at 80°C and 479 kPa. Several compounds are present in small amounts; however, volatiles such as 2-nonenal are minor but important flavor components in soybean oil.

The volatile profiles from the SFE soybean oil and the corresponding Tenax trap collected during the extraction of the oil at 80°C and 479 kPa, are compared in Fig 3. The mass spectra data of the volatiles indicated the compounds that were identified from the Tenax were typical of those found in the soybean oil (King *et al* 1988). Identification of the compounds is provided in Table 2. However, identified compounds listed in the Fig 3 caption were present only in the analyses of the Tenax traps and included acids, alcohols, ketones, and aromatic hydrocarbons. The second Tenax trap used to measure the breakthrough of volatiles indicated that only low molecular weight compounds C_2 and C_3 hydrocarbons and aldehydes were present at the conditions described in the experimental section.

The volatile compounds from the Tenax traps corresponded to those found in the oil sample as shown in Table 2 for each set of extraction conditions. The data represent two samples from two extractions and are the average of the four values. Total volatiles and individual peak areas from the oils extracted at 80°C and 479 kPa and 50°C and 383 kPa were much higher than the corresponding values from the Tenax traps. However, at the lower pressure of 96 kPa, there were smaller differences in the peak area of individual volatile compounds

TABLE 2
Volatiles[a] from SFE-soybean oil and from Tenax traps

Volatile compound	*Area* ($\times 10^3$)							
	80°C, 479 kPa		*50°C, 383 kPa*		*50°C, 96 kPa*		*240°C, 96 kPa*[b]	
	Oil[c]	*Tenax*[d]	*Oil*	*Tenax*	*Oil*	*Tenax*	*Oil*	*Tenax*
1. Ethanal	23·72	2·26	0·66	0·80	1·12	0·50	0·45	3·25
2. Pentane	66·43	4·59	6·43	3·83	3·49	6·84	23·80	23·99
3. Propanal	122·48	8·24	0·41	1·76	0·60	0·34	13·56	7·31
4. Ethanol	19·71	2·36	0·60	0·26	5·10	1·58	1·36	1·63
5. Butanal	15·33	1·48	0·24	0·32	0·73	0·65	0·65	0·15
6. 1-Propanol	61·54	4·16	2·47	1·45	0·44	0·25	0·26	1·23
7. 2-Ethylfuran	37·73	8·52	0·53	0·39	0·57	0·49	4·05	0·58
8. 2-Butenal	18·20	3·47	0·39	0·60	0·54	0·28	2·18	2·10
9. Pentanal	290·40	89·76	32·04	24·13	17·27	10·32	42·53	36·75
10. 2-Methylbutanal	27·75	25·86	1·95	1·22	0·40	0·83	8·78	3·48
11. 2-Pentenal	47·80	48·15	1·16	0·70	0·49	0·79	8·38	2·48
12. Pentanol	188·86	135·33	11·61	4·99	2·38	1·74	9·00	10·14
13. Hexanal	2982·03	533·32	213·53	146·37	25·29	25·12	122·66	120·93
14. 2-Hexenal	93·96	12·94	0·67	0·80	0·40	0·41	2·60	3·03
15. Hexanol	30·78	18·15	10·50	5·28	2·81	3·26	9·96	5·58
16. 2-Pentylfuran	32·07	3·91	0·52	2·53	0·72	1·01	6·03	2·56
17. 2-Heptenal	215·72	72·72	18·09	9·71	13·90	12·74	13·30	8·32
18. 1-Octen-3-ol	37·26	4·33	0·96	0·24	8·43	2·55	4·69	2·55
19. 2t,4c-Heptadienal	32·26	1·28	0·36	0·26	0·36	0·33	1·19	0·18
20. 2t,4t-Heptadienal	82·05	3·41	0·41	0·58	0·63	0·49	1·72	0·28
21. Hexanoic acid	103·59	23·52	0·64	0·50	0·57	0·67	1·73	0·20
22. 2-Octenal	21·04	2·10	0·42	0·22	0·19	0·49	1·42	0·10
23. Nonanal	39·01	2·15	0·77	0·51	1·14	0·24	0·52	0·36
24. 2t,4c-Decadienal	0·72	0·72	0·02	0·00	0·00	0·00	0·01	0·05
25. 2t,4t-Decadienal	0·92	2·47	0·39	0·00	0·00	0·02	0·15	0·30
Other	208·32	70·59	6·77	9·93	11·65	10·00	61·81	36·82

[a] Values for the volatile compounds are the average of two samples from different extractions.
[b] Volatiles from SFE-thermally treated soybean oil.
[c] Headspace volatile analysis of oil by dynamic headspace method.
[d] Thermal desorption of volatiles from Tenax.

in the oil and the traps. Total volatiles were greater in the oil samples at 240°C and 96 kPa than from the traps. Temperature has an effect on the formation of many volatiles (Selke and Frankel 1987); hexanal concentration was greater at the higher pressures when the temperature was 80°C. When soybean oil was treated at 240°C and 96 kPa in an attempt to degrade chromaphoric compounds, the amount of hexanal was much greater than when the extraction of soybeans was carried out 50°C and 96 kPa. High pressure tended to increase the hexanoic acid in the oil and Tenax analyses from extractions at 80°C and 479 kPa. Hexanal from the extraction at 80°C and 479 kPa was one compound that was greater in the Tenax sample than from the extracted oil.

The data from Table 2 were normalized to compare the relative amounts of each component obtained from the oil and from the Tenax traps (Table 3). The agreement between the two types of analyses in Table 3 was best at 50°C and 383 kPa, although good agreement was found at all three SFE conditions. The greatest difference in the volatile profiles occurs in the magnitude of the 2-heptenal peak for the extraction at 50°C and 96 kPa.

There was little difference in the relative values from the volatile analysis of the oils when the SFE was conducted at 50°C and 383 kPa or 80°C and 479 kPa as shown in Table 2. Hexanal was the most intense major peak varying from 49 to 68% of the total volatiles at the higher extraction pressures. However, at 96 kPa, hexanal was found to be only 25–30% of the total. Pentanal was present in relatively large quantities at all three extraction conditions. However, the pentanal concentration was lowest at 80°C and 479 kPa and increased as the pressure decreased. Heptenal accounted for 1–5% of the total components for the extraction at

TABLE 3
Volatiles[a] from SFE-soybean oil and from Tenax traps

Volatile compound	*Relative %*							
	80°C, 479 kPa		*50°C, 383 kPa*		*50°C, 96 kPa*		*240°C, 96 kPa*[b]	
	Oil[c]	*Tenax*[d]	*Oil*	*Tenax*	*Oil*	*Tenax*	*Oil*	*Tenax*
1. Ethanal	0·5	0·2	0·2	0·4	1·1	0·6	0·1	1·2
2. Pentane	1·4	0·4	2·1	1·8	3·5	8·3	7·0	8·7
3. Propanal	2·6	0·7	0·1	0·8	0·6	0·4	4·0	2·7
4. Ethanol	0·4	0·2	0·2	0·1	5·2	1·9	0·4	0·6
5. Butanal	0·3	0·1	0·1	0·1	0·7	0·8	0·2	0·1
6. 1-Propanol	1·3	0·3	0·8	0·7	0·4	0·3	0·1	0·4
7. 2-Ethylfuran	0·8	0·8	0·2	0·2	0·6	0·6	1·2	0·2
8. 2-Butenal	0·4	0·3	0·1	0·3	0·5	0·3	0·6	0·8
9. Pentanal	6·2	8·1	10·3	11·1	17·4	12·6	12·4	13·4
10. 2-Methylbutanal	0·7	2·3	0·6	0·6	0·4	1·0	2·6	1·3
11. 2-Pentenal	1·0	4·2	0·4	0·3	0·5	0·9	2·4	0·9
12. Pentanol	3·9	12·5	3·7	2·3	2·4	2·1	2·6	3·7
13. Hexanal	62·1	49·1	68·3	67·3	25·5	30·5	35·8	43·9
14. 2-Hexenal	2·0	1·1	0·2	0·4	0·4	0·5	0·8	1·1
15. Hexanol	0·7	1·5	3·4	2·4	2·8	3·9	2·9	2·0
16. 2-Pentylfuran	0·7	0·4	0·2	1·2	0·7	1·2	1·7	0·9
17. 2-Heptenal	4·6	6·8	5·8	4·5	14·0	15·4	3·9	3·0
18. 1-Octen-3-ol	0·8	0·4	0·3	0·1	8·5	3·1	1·4	1·0
19. 2t,4c-Heptadienal	0·6	0·1	0·1	0·1	0·4	0·4	0·5	0·1
20. 2t,4t-Heptadienal	1·3	0·3	0·1	0·2	0·6	0·5	0·5	0·1
21. Hexanoic acid	2·2	2·2	0·2	0·2	0·6	0·6	0·3	0·1
22. 2-Octenal	0·4	0·2	0·1	0·1	0·2	0·8	0·4	0·1
23. Nonanal	0·8	0·2	0·2	0·2	1·2	0·8	0·2	0·4
24. 2t,4c-Decadienal	0·1	0·1	0·0	0·0	0·0	0·0	0·0	0·0
25. 2t,4t-Decadienal	0·1	0·2	0·1	0·0	0·0	0·3	0·0	0·1
Other	4·3	6·5	2·2	4·6	11·8	12·2	18·0	13·2

[a] Values for the volatile compounds are the average of two samples from different extractions.
[b] Volatiles from SFE-thermally treated soybean oil.
[c] Headspace volatile analysis of oil by dynamic headspace method.
[d] Thermal desorption of volatiles from Tenax.

383 kPa and 479 kPa, and also from the SC-CO_2 conditioned oil, but was much higher (14–15%) in the samples extracted at 96 kPa.

The volatiles content of the oil treated at 240°C and 96 kPa and the corresponding Tenax trap which sampled the exhaust gas in this run are very similar in composition. It is interesting to note that the crude oil stock employed in this experiment contains a greater concentration of pentane than was found in the SFE oil. The presence of a large number of unidentified components in both the parent oil and Tenax trap may be related to the thermal degradation of components in the soybean oil at the high processing temperature. Such conditions were specifically chosen so as to thermally degrade color bodies that are present in the crude oil, resulting in an improved oil color.

Evaluation of crude oil by sensory and analytical methods has been reported to be a predictor of the quality of refined, bleached and deodorized soybean oil (Frankel *et al* 1988; Warner *et al* 1988). Oxidative damage of soybeans due to storage conditions and seed maturity has been monitored by the increase of volatile compounds in the crude oil (Frankel *et al* 1987). Analysis of volatiles collected on Tenax during SFE of soybean oil can be used as an indicator of oil quality during the SFE without removing an oil sample for analysis. The described sampling and analysis method could also be useful for monitoring the purity of CO_2 recycled in a reported semicontinuous SFE process (King *et al* 1988) for extracting soybeans. Such on-line monitoring could prevent recontamination of the soybean oil and extracted meal, thereby assuring a higher quality extract or residual meal for food use. Moreover, addition of a Tenax trap to the CO_2 exhaust port during the high pressure, thermal bleaching process can be used to characterize the volatiles generated from this process.

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